

CLAIMS:

[c01] 1. A method for preparing an alkali metal salt of a hydroxy-substituted hydrocarbon which comprises the steps of:

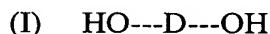
(i) contacting in a solvent media at least one hydroxy-substituted hydrocarbon with a base comprising an alkali metal cation; and

(ii) devolatilizing the solvent media comprising alkali metal salt by spraying the solvent media into a substantially water-immiscible organic solvent, said solvent being at a temperature greater than the boiling point of solvent media at the prevailing pressure.

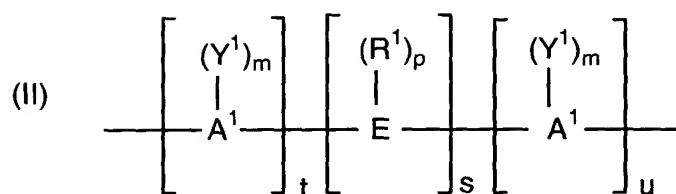
[c02] 2. The method of claim 1 wherein the hydroxy-substituted hydrocarbon is a hydroxy-substituted aromatic hydrocarbon selected from the group consisting of monohydroxy-substituted aromatic hydrocarbons; dihydroxy-substituted aromatic hydrocarbons; aromatic hydrocarbons substituted with multiple hydroxy groups; trihydroxy-substituted aromatic hydrocarbons; tetrahydroxy-substituted aromatic hydrocarbons; thiohydroxy-substituted aromatic hydrocarbons obtained by replacing one or more hydroxy groups of hydroxy-substituted aromatic hydrocarbons with thiohydroxy groups and mixtures of these substituted aromatic hydrocarbons.

[c03] 3. The method of claim 2 wherein the hydroxy-substituted aromatic hydrocarbon is at least one monohydroxy-substituted aromatic hydrocarbon selected from the group consisting of phenol, alkyl phenols, o-cresol, m-cresol, p-cresol, p-cumylphenol, resorcinol monomethyl ether, 1-naphthol, 2-naphthol, p-chlorophenol, o-chlorophenol and mixtures thereof.

[c04] 4. The method of claim 2 wherein the hydroxy-substituted aromatic hydrocarbon is at least one dihydroxy-substituted aromatic hydrocarbon of the formula (I):



wherein D has the structure of formula (II):



wherein A^1 represents an aromatic group;

E comprises a sulfur-containing linkage, sulfide, sulfoxide, sulfone; a phosphorus-containing linkage, phosphinyl, phosphonyl; an ether linkage; a carbonyl group; a tertiary nitrogen group; a silicon-containing linkage; silane; a cycloaliphatic group; cyclopentylidene, 3,3,5-trimethylcyclopentylidene, cyclohexylidene, 3,3-dimethylcyclohexylidene, 3,3,5-trimethylcyclohexylidene, methylcyclohexylidene, 2-[2.2.1]-bicycloheptylidene, neopentylidene, cyclopentadecylidene, cyclododecylidene, adamantylidene; an alkylene or alkylidene group, which group may optionally be part of one or more fused rings attached to one or more aromatic groups bearing one hydroxy substituent; an unsaturated alkylidene group; or two or more alkylene or alkylidene groups connected by a moiety different from alkylene or alkylidene and selected from the group consisting of an aromatic linkage, a tertiary nitrogen linkage; an ether linkage; a carbonyl linkage; a silicon-containing linkage, silane, siloxy; a sulfur-containing linkage, sulfide, sulfoxide, sulfone; a phosphorus-containing linkage, phosphinyl, and phosphonyl;

R^1 comprises hydrogen; a monovalent hydrocarbon group, alkenyl, allyl, alkyl, aryl, aralkyl, alkaryl, or cycloalkyl;

Y^1 independently at each occurrence is selected from the group consisting of an inorganic atom, a halogen; an inorganic group, a nitro group; an organic group, a monovalent hydrocarbon group, alkenyl, allyl, alkyl, aryl, aralkyl, alkaryl, cycloalkyl, and an alkoxy group;

the letter "m" represents any integer from and including zero through the number of positions on A^1 available for substitution;

the letter "p" represents an integer from and including zero through the number of positions on E available for substitution;

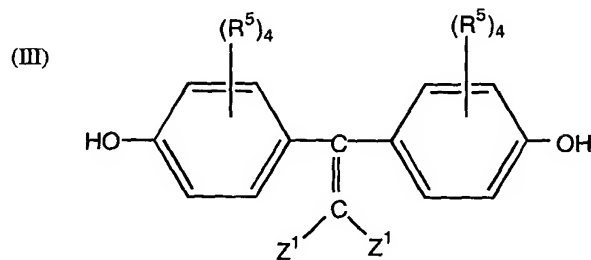
the letter "t" represents an integer equal to at least one;

the letter "s" represents an integer equal to either zero or one; and

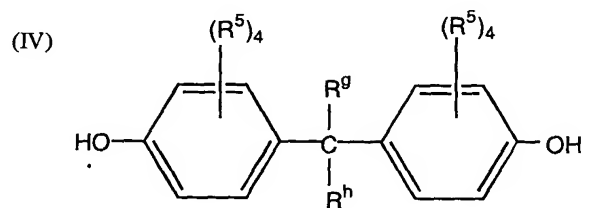
the letter "u" represents any integer including zero.

[c05] 5. The method of claim 2 wherein the hydroxy-substituted aromatic hydrocarbon is at least one dihydroxy-substituted aromatic hydrocarbon selected from the group consisting of 4,4'-(cyclopentylidene)diphenol; 4,4'-(3,3,5-trimethylcyclopentylidene)diphenol; 4,4'-(cyclohexylidene)diphenol; 4,4'-(3,3-dimethylcyclohexylidene)diphenol; 4,4'-(3,3,5-trimethylcyclohexylidene)diphenol; 4,4'-(methylcyclohexylidene)diphenol; 4,4'-bis(3,5-dimethyl)diphenol, 1,1-bis(4-hydroxy-3-methylphenyl)cyclohexane; 4,4-bis(4-hydroxyphenyl)heptane; 2,4'-dihydroxydiphenylmethane; bis(2-hydroxyphenyl)methane; bis(4-hydroxyphenyl)methane; bis(4-hydroxy-5-nitrophenyl)methane; bis(4-hydroxy-2,6-dimethyl-3-methoxyphenyl)methane; 1,1-bis(4-hydroxyphenyl)ethane; 1,2-bis(4-hydroxyphenyl)ethane; 1,1-bis(4-hydroxy-2-chlorophenyl)ethane; 2,2-bis(4-hydroxyphenyl)propane; 2,2-bis(3-phenyl-4-hydroxyphenyl)propane; 2,2-bis(4-hydroxy-3-methylphenyl)propane; 2,2-bis(4-hydroxy-3-ethylphenyl)propane; 2,2-bis(4-hydroxy-3-isopropylphenyl)propane; 2,2-bis(4-hydroxy-3,5-dimethylphenyl)propane; 3,5,3',5'-tetrachloro-4,4'-dihydroxyphenylpropane; bis(4-hydroxyphenyl)cyclohexylmethane; 2,2-bis(4-hydroxyphenyl)-1-phenylpropane; 2,4'-dihydroxyphenyl sulfone; dihydroxy naphthalene, 2,6-dihydroxy naphthalene; catechol; hydroquinone; resorcinol; C₁₋₃ alkyl-substituted resorcinols; 2,2-bis(4-hydroxyphenyl)butane; 2,2-bis(4-hydroxyphenyl)-2-methylbutane; 1,1-bis(4-hydroxyphenyl)cyclohexane; biphenol; bis(4-hydroxyphenyl); bis(4-hydroxyphenyl)ether; bis(4-hydroxyphenyl)sulfide; 2-(3-methyl-4-hydroxyphenyl)-2-(4-hydroxyphenyl)propane; 2-(3,5-dimethyl-4-hydroxyphenyl)-2-(4-hydroxyphenyl)propane; 2-(3-methyl-4-hydroxyphenyl)-2-(3,5-dimethyl-4-hydroxyphenyl)propane; bis(3,5-dimethylphenyl-4-hydroxyphenyl)methane; 1,1-bis(3,5-dimethylphenyl-4-hydroxyphenyl)ethane; 2,2-bis(3,5-dimethylphenyl-4-hydroxyphenyl)propane; 2,4-bis(3,5-dimethylphenyl-4-hydroxyphenyl)-2-methylbutane; 3,3-bis(3,5-dimethylphenyl-4-hydroxyphenyl)pentane; 1,1-bis(3,5-dimethylphenyl-4-hydroxyphenyl)cyclopentane; 1,1-bis(3,5-dimethylphenyl-4-hydroxyphenyl)cyclohexane; bis(3,5-dimethylphenyl-4-hydroxyphenyl)sulfide, 3-(4-hydroxyphenyl)-1,1,3-trimethylindan-5-ol, 1-(4-hydroxyphenyl)-1,3,3-trimethylindan-5-ol, 2,2,2',2'-tetrahydro-3,3,3',3'-tetramethyl-1,1'-spirobi[1H-indene]-6,6'-diol, and mixtures thereof, or wherein the hydroxy-substituted aromatic hydrocarbon is tris(4-hydroxyphenyl)ethane.

[c06] 6. The method of claim 2 wherein the hydroxy-substituted aromatic hydrocarbon is at least one dihydroxy-substituted aromatic hydrocarbon selected from the group consisting of those of the formula (III):



where independently each R⁵ is hydrogen, chlorine, bromine or a C₁₋₃₀ monovalent hydrocarbon or hydrocarbonoxy group, each Z¹ is hydrogen, chlorine or bromine, subject to the provision that at least one Z¹ is chlorine or bromine; and those of the formula (IV):



where independently each R⁵ is as defined hereinbefore, and R^g and R^h are each independently hydrogen or a C₁₋₃₀ hydrocarbon group.

[c07] 7. The method of claim 2 wherein the hydroxy-substituted aromatic hydrocarbon comprises bisphenol A.

[c08] 8. The method of claim 1 wherein the solvent media comprises water.

[c09] 9. The method of claim 8 wherein the amount of water removed during devolatilizing is greater than about 95 wt.%.

[c10] 10. The method of claim 8 wherein the amount of water removed during devolatilizing is greater than about 98 wt.%.

[c11] 11. The method of claim 8 wherein the amount of water removed during devolatilizing is greater than about 99 wt.%.

[c12] 12. The method of claim 8 wherein the solvent media further comprises at least one water-soluble protic organic solvent.

[c13] 13. The method of claim 12 wherein the water-soluble protic organic solvent comprises methanol or butanol.

[c14] 14. The method of claim 1 wherein the solvent media comprises at least one water-soluble protic organic solvent.

[c15] 14. The method of claim 1 wherein the alkali metal salt is essentially soluble in the solvent media.

[c16] 16. The method of claim 1 wherein the alkali metal cation is sodium.

[c17] 17. The method of claim 1 wherein the base is sodium hydroxide or sodium methoxide.

[c18] 18. The method of claim 1 wherein the amounts of hydroxy-substituted hydrocarbon and base in step (i) are stoichiometric, or deviate from stoichiometry by an amount in a range of between about 0.01 and about 1.2 mole %.

[c19] 19. The method of claim 1 wherein the amounts of hydroxy-substituted hydrocarbon and base in step (i) deviate from stoichiometry by no more than plus/minus 0.4 mole %.

[c20] 20. The method of claim 1 wherein the organic solvent is at least one member selected from the group consisting of alkylated benzenes, toluene, xylene, phenetole, anisole, veratrole, diphenylsulfone, halogenated benzenes, chlorinated benzenes, chlorobenzene, dichlorotoluene, dichlorobenzene, ortho-dichlorobenzene, para-dichlorobenzene, and 1,2,4-trichlorobenzene.

[c21] 21. The method of claim 1 wherein the organic solvent is toluene or ortho-dichlorobenzene.

[c22] 22. The method of claim 1 wherein the organic solvent is at a temperature in a range of between about 75°C and about 220°C.

[c23] 23. The method of claim 1 wherein the organic solvent is at a temperature in a range of between about 100°C and about 220°C.

[c24] 24. The method of claim 1 wherein the organic solvent is at a temperature in a range of between about 110°C and about 200°C.

[c25] 25. The method of claim 1 wherein the organic solvent is at a temperature in a range of between about 130°C and about 180°C.

[c26] 26. The method of claim 1 wherein the organic solvent is at a temperature in a range of between about 140°C and about 160°C.

[c27] 27. The method of claim 1 wherein the alkali metal salt is substantially insoluble in the organic solvent at the devolatilization temperature.

[c28] 28. The method of claim 1 wherein heat is provided to the organic solvent by circulating said solvent, optionally comprising alkali metal salt, through a pump-around loop comprising a centrifugal pump, and a spiral heat exchanger or a self-cleaning reboiler.

[c29] 29. The method of claim 1 wherein devolatilizing is performed in a vessel comprising subsurface baffles, at least one agitator and at least one spray nozzle for introduction of solvent media comprising alkali metal salt.

[c30] 30. The method of claim 29 wherein there is at least one gap between any baffle and the side of the vessel.

[c31] 31. The method of claim 1 further comprising at least one drying step after the devolatilizing step.

[c32] 32. The method of claim 31 wherein the drying step comprises distilling organic solvent and water from the vessel either with or without introduction of dry organic solvent to the vessel.

[c33] 33. The method of claim 31 wherein the amount of water remaining in the salt-containing organic solvent after one or more drying steps is less than about 100 ppm.

[c34] 34. The method of claim 31 wherein the amount of water remaining in the salt-containing organic solvent after one or more drying steps is less than about 20 ppm.

[c35] 35. The method of claim 31 wherein steps of devolatilizing and drying take place in one vessel.

[c36] 36. The method of claim 31 wherein the drying step takes place in a vessel different from the vessel used for the step of devolatilizing.

[c37] 37. The method of claim 1 wherein the alkali metal salt comprises less than 25% of particles with a diameter of greater than about 200 nm.

[c38] 38. The method of claim 1 wherein the alkali metal salt comprises less than 5% of particles with a diameter of greater than about 500 nm.

[c39] 39. The method of claim 1 wherein the alkali metal salt comprises less than 2% of particles with a diameter of greater than about 500 nm.

[c40] 40. The method of claim 1 further comprising at least one step of reducing the alkali metal salt particle size.

[c41] 41. The method of claim 40 wherein the particle size is reduced using equipment which comprises one or more of a centrifugal pump, grinder, drop-down blender, particle size reduction homogenizer or delumper.

[c42] 42. The method of claim 40 wherein the particle size reduction step is performed on a slurry of the alkali metal salt in organic solvent.

[c43] 43. The method of claim 1 wherein the alkali metal salt in organic solvent is at a solids level in a range of between about 5% and about 32% following devolatilizing.

[c44] 44. The method of claim 1 wherein the alkali metal salt in organic solvent is at a solids level in a range of between about 10% and about 20% following devolatilizing.

[c45] 45. A method for preparing a disodium salt of a dihydroxy-substituted aromatic hydrocarbon which comprises the steps of:

(i) contacting at least one dihydroxy-substituted aromatic hydrocarbon with sodium hydroxide in solvent media comprising water, wherein the amounts of dihydroxy-substituted aromatic hydrocarbon and sodium hydroxide deviate from stoichiometry by no more than plus/minus 0.4 mole %, and wherein the alkali metal salt is essentially soluble in the solvent media;

(ii) devolatilizing the solvent media comprising the disodium salt by spraying the solvent media into a substantially water-immiscible organic solvent in a vessel comprising subsurface baffles, at least one agitator and at least one spray nozzle for introduction of solvent media, wherein said organic solvent is at a temperature in a range of between about 110°C and about 200°C, wherein heat is provided to the organic solvent by circulating said solvent, optionally comprising disodium salt, through a pump-around loop comprising a centrifugal pump, and a spiral heat exchanger or a self-cleaning reboiler; wherein the amount of water removed during devolatilizing is greater than about 99 wt.%; and wherein said salt is at a solids level in a range of between about 10% and about 20% following devolatilizing; and

(iii) reducing the disodium salt particle size at least once in a slurry of the organic solvent.

[c46] 46. The method of claim 45 wherein the hydroxy-substituted aromatic hydrocarbon is bisphenol A.

[c47] 47. The method of claim 45 wherein the organic solvent is toluene or ortho-dichlorobenzene.

[c48] 48. The method of claim 47 wherein the organic solvent is ortho-dichlorobenzene.

[c49] 49. The method of claim 48 wherein the ortho-dichlorobenzene is at a temperature in a range of between about 130°C and about 180°C.

[c50] 50. The method of claim 45 wherein the particle size is reduced using equipment which comprises one or more of a centrifugal pump, grinder, drop-down blender, particle size reduction homogenizer or delumper.

[c51] 51. The method of claim 45 further comprising at least one drying step to remove residual water remaining after the devolatilizing step, wherein said drying step comprises distilling organic solvent and water from the vessel either with or without introduction of dry organic solvent to the vessel.

[c52] 52. The method of claim 51 wherein the amount of water remaining in the salt-containing organic solvent after one or more drying steps is less than about 20 ppm.

[c53] 53. The method of claim 51 wherein steps of devolatilizing and drying take place in one vessel.

[c54] 54. The method of claim 45 wherein the disodium salt comprises less than 25% of particles with a diameter of greater than about 200 nm; and less than 2% of particles with a diameter of greater than about 500 nm.

[c55] 55. A method for preparing the disodium salt of bisphenol A which comprises the steps of:

(i) contacting bisphenol A with sodium hydroxide in solvent media comprising water, wherein the amounts of bisphenol A and sodium hydroxide deviate from stoichiometry by no more than plus/minus 0.4 mole %;

(ii) devolatilizing the solvent media comprising the disodium salt by spraying the solvent media into ortho-dichlorobenzene in a vessel comprising subsurface baffles, at least one agitator and at least one spray nozzle for introduction of solvent media, wherein said ortho-dichlorobenzene is at a temperature in a range of between about 130°C and about 180°C, wherein heat is provided to the ortho-dichlorobenzene by circulating said ortho-dichlorobenzene, optionally comprising disodium salt, through a pump-around loop comprising a centrifugal pump, and a spiral heat exchanger or a self-cleaning reboiler; wherein the amount of water removed during devolatilizing is greater than about 99 wt.%; and wherein said salt is at a solids level in a range of between about 10% and about 20% following devolatilizing;

(iii) reducing the disodium salt particle size at least once in a slurry of ortho-dichlorobenzene to provide said disodium salt comprising less than 25% of particles with a diameter of greater than about 200 nm; and less than 2% of particles with a diameter of greater than about 500 nm, wherein the particle size is reduced using equipment which comprises one or more of a centrifugal pump, grinder, drop-down blender, particle size reduction homogenizer or delumper; and

(iv) drying at least once to remove residual water remaining after step (iii), wherein said drying step comprises distilling ortho-dichlorobenzene and water from the vessel either with or without introduction of dry ortho-dichlorobenzene to the vessel to provide said disodium salt with an amount of water remaining after one or more drying steps of less than about 20 ppm.

[c56] 56. A method for preparing an alkali metal salt of a hydroxy-substituted aromatic hydrocarbon which comprises the steps of:

(i) contacting at least one hydroxy-substituted aromatic hydrocarbon with a base in solvent media comprising water and at least one water-soluble protic organic solvent, wherein the amounts of hydroxy-substituted aromatic hydrocarbon and base deviate from stoichiometry by no more than plus/minus 0.4 mole %, and wherein the alkali metal salt is essentially soluble in the solvent media;

(ii) devolatilizing the solvent media comprising the salt by adding the solvent media into a substantially water-immiscible organic solvent in a vessel comprising subsurface baffles, at least one agitator and at least one inlet for introduction of solvent media, wherein said organic solvent is at a temperature in a range of between about 110°C and about 200°C, wherein heat is provided to the organic solvent by circulating said solvent, optionally comprising disodium salt, through a pump-around loop comprising a centrifugal pump, and a spiral heat exchanger or a self-cleaning reboiler; wherein the amount of water removed during devolatilizing is greater than about 99 wt.%; and wherein said salt is at a solids level in a range of between about 10% and about 20% following devolatilizing; and

(iii) reducing the salt particle size at least once in a slurry of the organic solvent.

[c57] 57. The method of claim 56 wherein the hydroxy-substituted aromatic hydrocarbon is a dihydroxy-substituted aromatic hydrocarbon.

[c58] 58. The method of claim 57 wherein the dihydroxy-substituted aromatic hydrocarbon is bisphenol A.

[c59] 59. The method of claim 56 wherein the alkali metal cation is sodium.

[c60] 60. The method of claim 56 wherein the base is sodium hydroxide.

[c61] 61. The method of claim 56 wherein the water-soluble protic organic solvent comprises methanol.

[c62] 62. The method of claim 56 wherein the organic solvent is toluene or ortho-dichlorobenzene.

[c63] 63. The method of claim 62 wherein the organic solvent is ortho-dichlorobenzene.

[c64] 64. The method of claim 63 wherein the ortho-dichlorobenzene is at a temperature in a range of between about 130°C and about 180°C.

[c65] 65. The method of claim 56 wherein the particle size is reduced using equipment which comprises one or more of a centrifugal pump, grinder, drop-down blender, particle size reduction homogenizer or delumper.

[c66] 66. The method of claim 56 further comprising at least one drying step to remove residual water remaining after the devolatilizing step, wherein said drying step comprises distilling a mixture comprising organic solvent and water from the vessel either with or without introduction of dry organic solvent to the vessel.

[c67] 67. The method of claim 66 wherein the amount of water remaining in the salt-containing organic solvent after one or more drying steps is less than about 20 ppm.

[c68] 68. The method of claim 67 wherein steps of devolatilizing and drying take place in one vessel.

[c69] 69. The method of claim 56 wherein the salt comprises less than 25% of particles with a diameter of greater than about 200 nm; and less than 2% of particles with a diameter of greater than about 500 nm.

[c70] 70. A method for preparing the disodium salt of bisphenol A which comprises the steps of:

(i) contacting bisphenol A with sodium hydroxide in solvent media comprising water and methanol, wherein the amounts of bisphenol A and sodium hydroxide deviate from stoichiometry by no more than plus/minus 0.4 mole %;

(ii) devolatilizing the solvent media comprising the disodium salt by adding the solvent media to ortho-dichlorobenzene in a vessel comprising subsurface baffles, at least one agitator and at least one inlet for introduction of solvent media, wherein said ortho-dichlorobenzene is at a temperature in a range of between about 130°C and about 180°C, wherein heat is provided to the ortho-dichlorobenzene by circulating said ortho-dichlorobenzene, optionally comprising disodium salt, through a pump-around loop comprising a centrifugal pump, and a spiral heat exchanger or a self-cleaning reboiler; wherein the amount of water removed during devolatilizing is greater than about 99 wt.%; and wherein said salt is at a solids level in a range of between about 10% and about 20% following devolatilizing;

(iii) reducing the disodium salt particle size at least once in a slurry of ortho-dichlorobenzene to provide said disodium salt comprising less than 25% of particles with a diameter of greater than about 200 nm; and less than 2% of particles with a diameter of greater than about 500 nm, wherein the particle size is reduced using equipment which comprises one or more of a centrifugal pump, grinder, drop-down blender, particle size reduction homogenizer or delumper; and

(iv) drying at least once to remove residual water remaining after step (iii), wherein said drying step comprises distilling ortho-dichlorobenzene and water from the vessel either with or without introduction of dry ortho-dichlorobenzene to the vessel to provide said disodium salt with an amount of water remaining after one or more drying steps of less than about 20 ppm.